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JP55063682A

(58) Field of Search

UK CL (Edition P) A5E EN ES, C4X

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(54) Abstract Title

House dust mite allergen deactivation

(57) A Der-f allergen is deactivated by contacting it with a deactivating effective amount of one or more of

- i) urea,
- ii) cedarwood oil,
- iii) cyclodextrin,
- iv) hexadecyltrimethylammonium chloride
- v) aluminium chlorohydrate,
- vi) 1-propoxy-propanol-2,
- vii) polyquaternium-10
- viii) silica gel,
- ix) hydrogenated hop oil,
- x) propylene glycol alginate,
- xi) polyvinyl pyrrolidone,
- xii) ammonium sulphate,
- xiii) hinokitiol,
- xiv) N-methyl pyrrolidone,
- xv) L-ascorbic acid,
- xvi) "immobilised tannic acid",
- xvii) chlorohexidine,
- xviii) maleic anhydride,
- xix) the sodium salt of anthraquinone,
- xx) hinoki oil,
- xxi) a composite of silver chloride and TiO₂
- xxii) diazolidinyl urea,
- xxiii) 6-isopropyl-m-cresol,
- xxiv - xvi) a compound of formula I or II herein or a polymeric compound containing two or more of a

recurring unit of the formula III herein.

The deactivant may be combined with a propellant and optionally a solvent to give an aerosol composition.

GB 2 329 586 A

Improvements in or relating to organic compositions

5 It has been known for a long time that house dust
can trigger allergenic reactions in humans, such as
asthma and rhinitis. It was reported, as early as
1928, that it was the dust mites in the dust that were
the primary source of the allergenic response but it
was only in the 60's that researchers appreciated its
significance.

10 It is believed that the faeces of the house dust
mite, *Dermatophagoides farinae* (known as Der-f) and
Dermatophagoides pteronyssinus (known as Der-p) trigger
the immune responses of the body, thereby giving rise
to well known allergenic symptoms.

15 A review of this is given in **Experimental and
Applied Acarology**, 10 (1991) p. 167-186 in an article
entitled "House dust-mite allergen" : A review by L. G.
Arlian.

20 One way to overcome these allergenic response has
been to thoroughly vacuum surfaces, such as carpets,
that contain the dust mites and their faeces thoroughly
and often, but that is both time consuming (i.e. has to
be regularly done if one wants to make an allergenic
25 free environment) and is very dependant on the
efficiency of vacuum cleaner and filter bag used e.g.
micron filter bag or 2 layer vacuum bags.

30 An alternative method of creating an allergen-free
environment has been to denature the allergen, for

example as described in US Patent No. 4,806,526. The only effective method however of which we are aware is to apply tannic acid to the allergen. However, tannic acid can cause staining, and this is a particularly acute problem for light coloured carpets (e.g. white and light beige carpets) and other textile surfaces as tannic acid leaves a deep brown stain.

Therefore, we have been looking for allergenic denaturants which will not stain susceptible surfaces such as carpets and still deactivate the allergen.

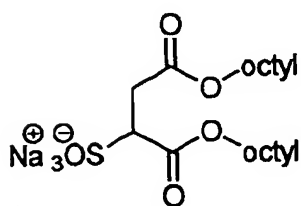
We have surprisingly found that deactivants are specific to the type of dust mite allergen being treated. For example an effective Der-f allergen deactivants will not automatically work effectively as a Der-p allergen deactivant.

We have looked into Der-f allergen deactivant and have found only a select number of deactivants destroy the Der-f allergen, whilst at the same time not leaving a stain.

According to the invention there is provided a method for deactivating a Der-f allergen comprising contacting the allergen with a deactivating effective amount of one or more of deactivants (herein after defined as the deactivant) selected from

- i) urea,
- ii) cedarwood oil,
- iii) cyclodextrin,
- iv) hexadecyltrimethylammonium chloride
- v) aluminium chlorohydrate,

- vi) 1-propoxy-propanol-2,
vii) polyquaternium-10
viii) silica gel ,
ix) hydrogenated hop oil,
x) propylene glycol alginate,
5 xi) polyvinyl pyrrolidone,
xii) ammonium sulphate,
xiii) hinokitiol,
xiv) N-methyl pyrrolidone,
xv) L-ascorbic acid,
10 xvi) "immobilised tannic acid", (hereinafter
defined)
xvii) chlorohexidine,
xviii) maleic anhydride,
xix) the sodium salt of anthraquinone,
xx) hinoki oil,
15 xxi) a composite of AgCl and TiO₂
xxii) diazolidinyl urea,
xxiii) 6-isopropyl-m-cresol,
xxiv) a compound of formula I,

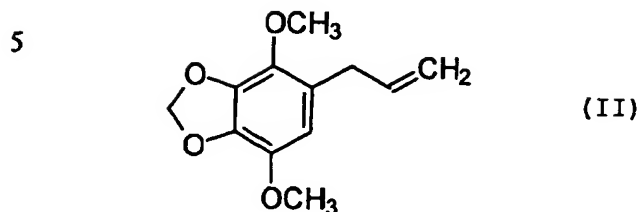


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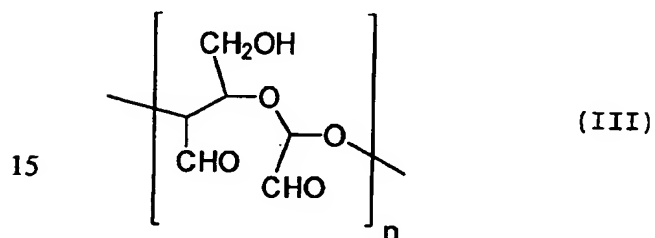
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xxv) a compound of formula II



xxvi) a polymeric compound containing two or more of
10 a recurring unit of the formula III



where $n = 2$ to 200.

20 In this Specification, the definition of the following compounds or compositions is given below:
A compound of formula I is commercially available as Aerosol OT.

25 A compound of formula II is commercially available as parsley camphor.

Hinoki oil is a mixture of Thujan-3-one, 2-pinene, 3,5,7,3',4' pentahydroflavanone and 1,3,3,-trimethyl-2-norcamphanone.

Cedarwood oil, contains α - and β - cedrene (ca 80%), cedrol (3-14%) and cedrenol. Other sesquiterpenes and some monoterpenes are also present.

5 Polyquaternium-10 is a polymeric quaternary ammonium salt of hydroxy ethyl cellulose reacted with a trimethyl ammonium substituted epoxide commercially available as Polymer JR-125.

10 Silica gel is also known as colloidal silica or silicic acid and is commercially available as Kent.

Hinokitiol is also known as β -thujaplicin.

"Immobilised tannic acid" is tannic acid on
15 polyvinyl pyrrolidone beads. "Immobilised Tannic Acid" is prepared as follows:

100 mg of tannic acid dissolved in water, 50 mg of Polyclar 10 (ISP, Guildford Surrey) polyvinyl pyrrolidone beads were added and stirred for one hour.
20 The beads were filtered off the solution and washed with a few mls of iced water until no colour was seen in the washings. They were then dried in the oven at 50°C.

25 The composite of silver chloride and TiO_2 is made up of 20% wt/wt AgCl on 80% TiO_2 3-5 μm porous beads.

The deactivant is present in an amount of from 0.01
- 7%, preferably 0.01 to 3%.

Preferably the amount of deactivant present in such a method is from 0.5 oz to 5 oz per 10 yds², more preferably 1 oz per 11 yds² of area treated.

5 Preferably the deactivant is selected from

hinoki oil,
a composite of AgCl and TiO₂,
diazolidinyl urea
6-isopropyl-m-cresol,
10 chlorohexidine,
maleic anhydride,
the sodium salt of anthraquinone and
a compound of formula I or II, defined above.

15 Further according to the invention there is provided an aerosol composition containing

a) a deactivant (hereinafter the Deactivant) selected from

- 20 i) urea,
ii) cedarwood oil,
iii) cyclodextrin,
iv) hexadecyltrimethylammonium chloride
v) aluminium chlorohydrate,
vi) 1-propoxy-propanol-2,
vii) polyquaternium-10,
25 viii) silica gel ,
ix) hydrogenated hop oil,
x) propylene glycol alginate,
xi) polyvinyl pyrrolidone,
xii) ammonium sulphate,
xiii) hinokitiol,
30 xiv) N-methyl pyrrolidone,

- xv) L-ascorbic acid,
xvi) "immobilised tannic acid", (hereinafter defined)
xvii) chlorohexidine,
xviii) maleic anhydride,
5 xix) the sodium salt of anthraquinone,
xx) hinoki oil,
xxi) a composite of silver chloride and TiO_2 ,
xxii) diazolidinyl urea,
xxiii) 6-isopropyl-m-cresol,
10 xxiv-xxvi) a compound of formula I, II or III defined above;

b) a propellant and

15 c) optionally a solvent.

Preferably the amount of deactivant present in such a composition is from 0.01 - 7%, preferably 0.01 to 3%,

20 Preferably the amount of propellant present in such a composition is 4-50%, more preferably 4 to 30%,

Preferably the amount of solvent present in such a composition is 0 to 99.95, more preferably 0 to 90%, most preferably 20 to 90%.

25 Preferably the deactivant is selected from

hinoki oil,
a composite of AgCl with TiO_2 ,
diazolidinyl urea,
30 6-isopropyl-m-cresol,
chlorohexidine,

maleic anhydride,
the sodium salt of anthraquinone and
a compound of formula I or II defined above.

5 Preferably the propellant is selected from those commercially available, for example C_{1-4} alkanes and hydrochlorofluorocabrons and compressed gases such as nitrogen air and carbon dioxide.

10 Preferably the solvent is selected from C_{1-6} alcohols (e.g. ethanol) or water.

In addition the composition may also contain one or more of the following

15 a fragrance, (preferably in an amount of 0 to 5%), more preferably 0 to 2%.

an antimicrobial compound e.g. alkyl dimethyl benzyl ammonium saccharinate (preferably in an amount of 0.01 to 1%)

20 a surfactant (e.g. Dow Corning 193 Surfactant or (preferably in an amount of 0.01 to 1%)

25 a corrosion inhibitor (e.g. sodium nitrite, sodium benzoate, triethanolamine and ammonium hydroxide (preferably in an amount of 0.01 to 10%), and/or

30 (a miticide) (such as benzyl benzoate, pyrethroid pemethrin, d-allethrin and optionally a synergist such as pipernoil butoxide (preferably in an amount of 0.1 to 10%)).

It has been found that deactivants of the invention have as effective allergen deactivating properties as tannic acid but without the drawback of staining.

5 The invention will now be illustrated by the following Examples.

The test procedure in Examples 1 to 17 is as follows and is known as the ELISA protocol.

10 The ELISA protocol for Der-f has been developed as follows as a measure of denaturing property for denaturants.

ELISA Protocol 1

15

1. Dust is collected from Hoover (a trademark) bags and passed through a series of sieves down to 63 microns.

20

2. Clean petri dishes are labelled with the chemical to be tested (on the base), three replicates are used for each treatment.

25

3. Filter paper is used to line each dish and 0.2g of dust is added to each dish onto the filter paper. The lid (or base, as dishes are inverted) is replaced and the dish is shaken to disperse of dust evenly over the filter paper.

30

4. 2% aqueous solutions of deactivant was used except for the silver chloride composite where 0.05% was used instead. Immobilised tannic acid was used as a 1%

dispersion. The hydrogenated hop end was used at the 2% level (in the form of a 10% solution). Water insoluble deactivant were emulsified with surfactant (a sorbitone oleate surfactant (i.e. Tween). Hinokitol was used at 0.5% not 2%.

5

5. The dust is sprayed with the corresponding treatment, 2 sprays are required for sufficient coverage (1 spray = 1.5ml).

10

6. Leave uncovered at room temperature, in well aerated room, until filter paper is dry. This can take up to 4 hours.

15

7. Empty dust in epindorfs labelled according to treatment.

20

8. Add 1 ml of 5% Bovine Serum Albumen Phosphate Butter Saline - Tween BSA-PBS-T to each epindorf (5 times the weight of dust) (20ml of BSA-PBS-T = 1g of BSA in 20ml of PBS-T).

25

9. Leave overnight in the fridge.

10. Centrifuge for 5 minutes at 13,000 rpm.

30

11. Decant the supernatant into a new epindorf labelled according to treatment.

12. Centrifuge again for 5 minutes at 13,000 rpm.

13. Make up dilution's of 1:10 and 1:100 by adding 100ul of neat solution to 900ul of 1% BSA-PBS-T (1:10).

30

This is repeated using 100ul of 1:10 dilution and add to 900ul of 1% BSA-PBS-T for 1:100 dilution.

ELISA Protocol 2 for Der f 1: Indoor Biotechnologies

- 5 1. Prepare samples and dilutions as in protocol 1.
2. Prepare 500 ml of 50 mM carbonate/bicarbonate buffer by dissolving 0.795g Na_2CO_3 and 1.465g NaHCO_3 in 500ml of distilled water. Check the pH is at 9.6.
10 (This solution is kept in the fridge in a conical flask).
3. Monoclonal antibody, this is kept in the freezer. (1µg per well ; 11ml is needed) has to be added to the buffer using the following method this is
15 applied to the ELISA plate:
 - 11ml of carbonate/bicarbonate buffer is added to the dispensing tray.
 - 11µl of Der f 1 monoclonal antibody (Stored in freezer, epindorf in use is in
20 the fridge) is added to the buffer. To ensure that all the antibody is removed from the tip, wash out the pipette tip by sucking up and down I the buffer solution, gently stirring to mix thoroughly.
- 25 4. Pipette 100µl of the antibody solution into each well of the microtiter plate, cover with a plate sealer and leave overnight at 4°C.
- 30 5. Empty the plate by quickly inverting it over the sink, then dry by banging on a stack of paper

towels.

6. Add 200 μ l of wash buffer to each well: PBS/0/05% tween (PBS-T).

5 7. Repeat stages 5 and 6 once more (making a total of 2 washes).

8. Make sure all the wells are dry, then add 100 μ l of 1% BSA-PBS-T. Replace the plate sealer and incubate
10 for 1 hour at room temperature*.

9. Repeat steps 5 to 7 (2 washes).

10. *During the hour incubation period, prepare the
15 allergen standards at dilutions between 125 and 1 ng/ml Der f 1:

- Add 25 μ l of allergen standard (kept in the fridge in polystyrene box) to 475 μ l of 1% PBS-BSA-T and mix thoroughly - labelled '125'.

20 - 250 μ l of 1% PBS-BSA-T is added 7 further epindorfs which are labelled 62.5, 31.25, 15.63, 7.61, 3.9, 1.95 and 0.98.

- 250 μ l is taken from the 1st epindorf (labelled 125) and transferred to the next (labelled 62.5). This is mixed thoroughly.

25 - Using a new pipette tip, 250 μ l is removed from epindorf labelled 62.5 and transferred to 31.25, this procedure is continued down to the 0.98 concentration (125, 62.5, 31.25, 15.63, 7.61, 3.9, 1.95, 0.98)

30 - In total 475+(250x7)=2.3ml : 0.023g of BSA

added to 2.3ml of PBS-T.

- 5 11. Add 100 μ l aliquots of the allergen sample to the plate along with the standard allergen samples for the reference curve in duplicate. The standards usually go in the first two columns on the left hand side, with the least concentrated on top. Incubate for 1 hour.
- 10 12. Follow stages 5 to 6, completing a total of 5 washes.
- 15 13. Pore 11ml of 1% BSA-PBS-T (0.11g of BSA to 11ml of PBS-T) to the dispensing tray. Add 11 μ l of the biotinylated monoclonal antibody (fridge) and mix thoroughly.
14. Pipette 100 μ l into each well and incubate for 1 hour at room temperature.
- 20 15. Empty plate and wash as described in stage 12. (5 washes).
- 25 16. Add 11 μ l of Streptavidin (freezer) to 11ml of 1% BSA-PBS-T. Pipette 100 μ l into each well and incubate for 30 minutes. Reserve any remaining solution in a vial.
17. Empty plate and wash as described in stage 12 (5 washes).
- 30 18. Make a solution of OPD, by putting the two tablets (in silver and gold foil) into 20 ml of distilled

water (in a glass vial). Shake quite vigorously in the dark until the tablets have dissolved (Wrap the vial up either in tin foil or paper towel).

5 19. Add a small amount to the remaining solution from stage 16. Wait for a colour change (positive reaction). Add 200 μ l to each well and incubate for a minimum of 30 minutes in the dark.

10 20. Read the plate at 450nm/405nm if filter not available.

Examples 1 to 26

15 The deactivants, as set out in the following table, were treated according to the above procedure and the results are as given below. Tannic acid was used as a comparator. What was measured after treatment with deactivation tannic acid was the amount of allergen remaining active after treatment. The ratio of amount of remaining active allergen after treatment with
20 deactivant tannic acid is also given.

25

30

Table

Example	Deactivant	Amount of Allergen remaining active after deactivant treatment	Amount of Allergen remaining active after tannic acid treatment	Ratio of remaining active allergen after Deactivant/Tannic Acid Treatment	Number
1	Urea	3750	1500	2.500	i
2	Polymetric dialdehyde	1325	550	2.409	xxvi
3	Cedarwood oil	1800	750	2.400	ii
4	Cyclodextrin	3850	1700	2.265	iii
5	hexadecyltrimethylammonium chloride	4075	1800	2.264	iv
6	Aluminium chlorohydrate	1675	750	2.233	v
7	1-propoxy-propanol-2	3950	1800	2.194	vi
8	Silica Gel (Kent)	2037.5	933.5	2.183	viii
9	polyquaternium-10 (Polymer JR-125)	4335	2000.00	2.168	vii
10	Hydrogenated Hop Oil	1100	550	2.000	ix
11	Propylene glycol alginate	3175	1700	1.868	x
12	Poly vinyl pyrrolidone	2450	1425	1.719	xi
13	Ammonium sulphate	2750	1700	1.618	xii

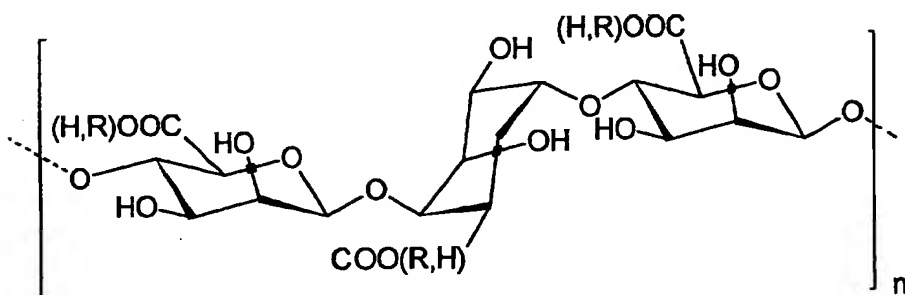
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In the table certain compounds are used that are defined as follows:

Hydrogenated Hop Oil is the potassium salt of tetrahydroiso humulinic acid (known as reduced isomerised hop extract).

Polymeric dialdehyde is a compound containing 2-200 recurring units of the formula III.

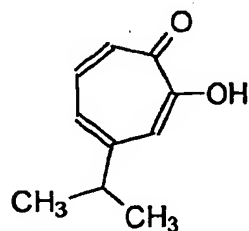
Propylene glycol alginate is



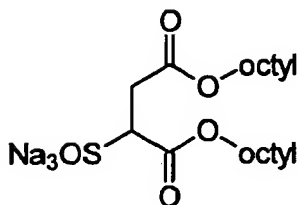
Chlorohexadene is 1,1'-hexamethylene bis

[5-(4-chlorophenyl)-biguanide]

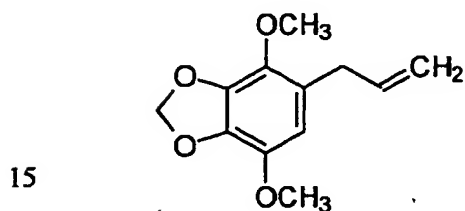
Hinokitiol is β -thujaplin, a compound of the formula



Aerosol OT is a compound of the formula



10 Parsley extract is a compound of the formula



20 Hinoki oil is a mixture of Thujan-3-one, 2-pinene, 3,5,7,3',4' pentahydroflavanone and 1,3,3, - trimethyl-2-norcamphanone.

Germall II is diazolidinyl urea and
Thymol is 6-isopropyl -m- cresol

Examples 27 to 30

25 The following formulations can be made up as a compositions for use as an aerosol for deactivating der-f allergens).

EXAMPLE 27

<u>Raw Ingredient Description</u> <u>By Weight</u>		<u>Item Classification</u>	<u>%</u>
5	Anhydrous Ethanol (SD Alcohol 40)	Solvent	79.646
	Alkyl dimethyl benzyl ammonium saccharinate	Cationic Surfactant	0.106
	Corrosion Inhibitor		0.192
10	Corrosion Inhibitor		0.192
	Corrosion Inhibitor		0.096
	Deionized Water	Water/Solvent	15.768
15	Carbon Dioxide	Propellant	4.000
	TOTAL		100.000

20

25

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EXAMPLE 28

	<u>Raw Ingredient</u> <u>Description by Weight</u>	<u>Item Classification</u>	<u>%</u>
5	Anhydrous Ethanol (SD Alcohol 40)	Solvent	* 57.000
	Fragrance#17	Fragrance	0.0500
	Dow Corning 193 Surfactant	Surfactant	0.025
10	Corrosion Inhibitor		0.100
	Corrosion Inhibitor		0.100
	Deionized Water	Water/solvent	* 14.725
15	NP-40/Butane 40	Hydrocarbon propellant	28.000
	TOTAL		100.000

* = May replace with 95% Ethanol (SD Alcohol 40) at 61.755% by weight and 9.970% by weight Deionized water

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EXAMPLE 29

	<u>Raw Ingredient</u> <u>Description by Weight</u>	<u>Item Classification</u>	<u>%</u>
5	Anhydrous Ethanol (SD Alcohol 40)	Solvent	79.646
	Benzyl Benzoate - an acaricide	Active/ester	4.600
10	Alkyl dimethyl benzyl ammonium saccharinate	Cationic Surfactant	0.106
	Corrosion Inhibitor		0.192
	Corrosion Inhibitor		0.192
	Corrosion Inhibitor		0.096
15	Deionized Water	Water/solvent	11.168
	Carbon Dioxide	Propellant	4.000
	TOTAL		100.000

20

25

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EXAMPLE 30

	<u>Raw Ingredient</u> <u>Description by weight</u>	<u>Item Classification</u>	<u>%</u>
5	Anhydrous Ethanol (SD Alcohol 40)	Solvent	*57.000
	Benzyl Benzoate	Active/ester	4.600
	Fragrance#17	Fragrance	0.0500
10	Dow Corning 193 Surfactant	Surfactant	0.025
	Corrosion Inhibitor		0.100
	Corrosion Inhibitor		0.100
15	Deionized Water	Water/solvent	*10.125
	NP-40/Butane 40	Hydrocarbon propellant	28.000
	TOTAL		100.000

20 * = May replace 95% Ethanol (SD Alcohol 40) at 61.755%
by weight and 5.370% by weight Deionized water.

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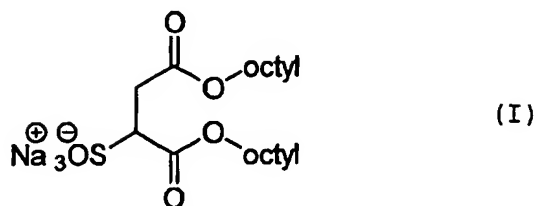
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CLAIMS

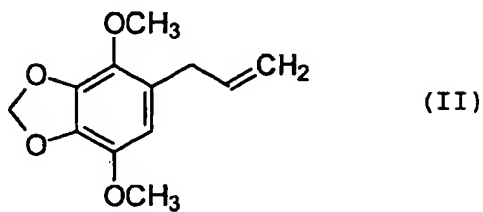
1. A method for deactivating a Der-f allergen comprising contacting the allergen with a deactivating effective amount of one or more of deactivants (herein
5 after defined as the Deactivant) selected from

- i) urea,
- ii) cedarwood oil,
- iii) cyclodextrin,
- 10 iv) hexadecyltrimethylammonium chloride
- v) aluminium chlorohydrate,
- vi) 1-propoxy-propanol-2,
- vii) polyquaternium-10
- viii) silica gel ,
- ix) hydrogenated hop oil,
- 15 x) propylene glycol alginate,
- xi) polyvinyl pyrrolidone,
- xii) ammonium sulphate,
- xiii) hinokitiol,
- xiv) N-methyl pyrrolidone,
- 20 xv) L-ascorbic acid,
- xvi) "immobilised tannic acid", (hereinafter defined)
- xvii) chlorohexidine,
- xviii) maleic anhydride,
- xix) the sodium salt of anthraquinone,
- 25 xx) hinoki oil,
- xx) a composite of silver chloride and TiO_2 ,
- xxi) diazolidinyl urea,
- xxii) 6-isopropyl-m-cresol,

xxiv) a compound of formula I,

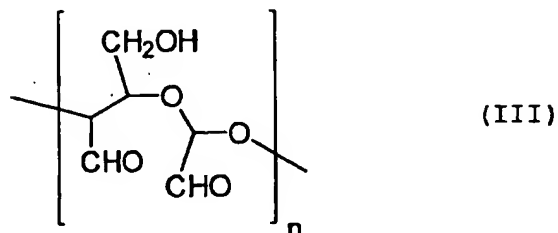


xxv) a compound of formula II



and

xxvi) a polymeric compound containing two or more of
a recurring unit of the formula III



where $n = 2$ to 200

2. A method according to Claim 1 in which the amount of Deactivant present is from 0.5 oz to 5 oz per 10 yds?

3. A method according to Claim 1 or Claim 2 in which the Deactivant is selected from

5

hinoki oil,
a composite of AgCl with TiO₂,
diazolidinyl urea
6-isopropyl-m-cresol,
10 chlorohexidine,
maleic anhydride,
the sodium salt of anthraquinone and
a compound of formula I or II, defined in Claim 1.

4. An aerosol composition containing

15

a) a deactivant selected from

20

- i) urea,
- ii) cedarwood oil,
- iii) cyclodextrin,
- iv) hexadecyltrimethylammonium chloride
- v) aluminium chlorohydrate,
- vi) 1-propoxy-propanol-2,
- vii) polyquaternium-10
- viii) silica gel ,
- 25 ix) hydrogenated hop oil,
- x) propylene glycol alginate,
- xi) polyvinyl pyrrolidone,
- xii) ammonium sulphate,
- xiii) hinokitiol,
- 30 xiv) N-methyl pyrrolidone,
- xv) L-ascorbic acid,

xvi) "immobilised tannic acid", (hereinafter defined)

xvii) chlorohexidine,

xviii) maleic anhydride,

xix) the sodium salt of anthraquinone,

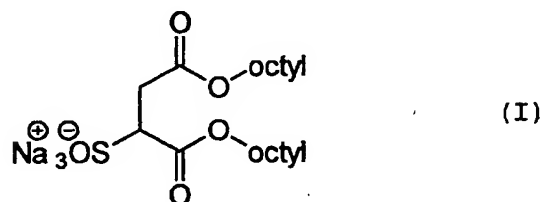
xx) hinoki oil,

xxi) a composite of silver chloride and TiO_2

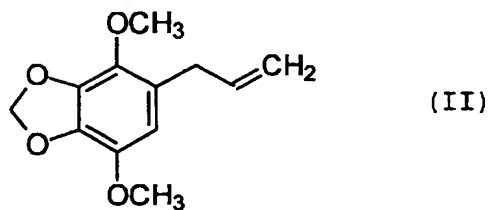
xxii) diazolidinyl urea,

xxiii) 6-isopropyl-m-cresol,

xxiv a compound of formula I,



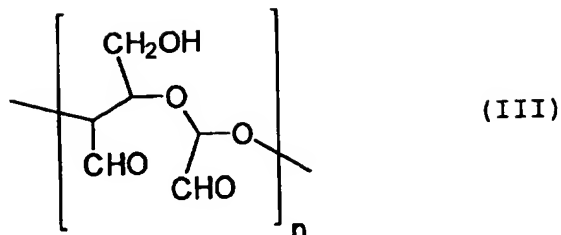
xxv) a compound of formula II



and

xxiv) a polymeric compound containing two or more of a recurring unit of the formula III

5



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where $n = 2$ to 200 (hereinafter defined as the Deactivant).

b) a propellant and

c) optionally a solvent.

15

6. A composition according to Claim 5 in which the amount of Deactivant present in such a composition is from 0.01 to 7%, the amount of propellant present in such a composition is 0.05 to 3%, and the amount of solvent present in such a composition is 0 to 99.95%.

20

7. A composition according to Claim 5 or Claim 6 in which the Deactivant is selected from

hinoki oil,

a composite of AgCl with TiO_2 ,

diazolidinyl urea,

25

6-isopropyl-m-cresol,

chlorohexidine,

maleic anhydride,

the sodium salt of anthraquinone and

a compound of formula I or II defined above.

30

8. A composition according to any one of Claims 4 to 7 in which the propellant is selected from C₁₋₄ alkane and carbon dioxide.

5 9. A composition according to any one of Claims 4 to 8 in which the solvent is selected from C₁₋₆ alcohols (e.g. ethanol) or water.

10 10. A composition according to any one of Claims 4 to 9 in which the composition may also contain one or more of the following

a fragrance,

a surfactant (e.g. Dow Corning 193 Surfactant

an antimicrobial agent (e.g. alkyl dimethyl benzyl ammonium saccharinate),

15 a corrosion inhibitor (e.g. sodium nitrite, sodium benzoate, triethanolamine and ammonium hydroxide), and/or

a miticide (such as benzyl benzoate).

20 11. A method for denaturing a Der-f allergen substantially as herein described with reference to any one of the Examples.

12. A composition substantially as herein described with reference to any one of the Examples.

25

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The
Patent
Office

2^a

Application No: GB 9720275.8
Claims searched: 1-3 and 11

Examiner: Peter Davey
Date of search: 14 January 1998

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.P): A5E (EN)

Int Cl (Ed.6): A01N

Other: Online: WPI, CAS ONLINE

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	GB 2300122 A (SINCLAIR), see eg. claims 1 and 3	1 at least
A	WO 96/09762 A1 (LABS. PHYTODIFF), see eg. claim 1	1 at least

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.

& Member of the same patent family

A Document indicating technological background and/or state of the art:
P Document published on or after the declared priority date but before the filing date of this invention.
E Patent document published on or after, but with priority date earlier than, the filing date of this application.



Application No: GB 9720275.8
Claims searched: 4-10 and 12

Examiner: Peter Davey
Date of search: 8 April 1998

Patents Act 1977
Further Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.P): A5E (ES), C4X

Int CI (Ed.6): A01N 25/06, C09K 3/30

Other: Online: WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	GB 1397216 (COLGATE-PALMOLIVE), see eg. claim 1 and Example	4 at least
X	GB1167173 (SPITZER & SMALL), see eg. Exs. 1, 2, 5 and 6	4 at least
X	WO 96/04937 A1 (PROCTER & GAMBLE), see eg. claims 1 and 6	4 at least
X	US 5415815 (BRUNO), see eg. claim 1	4 at least
X	US 4752466 (JOHNSON & JOHNSON), see eg. claim 2	4 at least
X	US 4715387 (UNIV. OF CALIFORNIA), see eg. claim 3	4 at least
X	WPI Abstract Acc. No. 80-48710C/198028 and JP 55069682 A (TOYO AEROSOL), see abstract	4 at least

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.

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A Document indicating technological background and/or state of the art
P Document published on or after the declared priority date but before the filing date of this invention.
E Patent document published on or after, but with priority date earlier than, the filing date of this application.